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A PARTICLE CLASS BALANCE RECEPTOR MODEL FOR AEROSOL APPORTIONMENT IN SYRACUSE, N.Y.

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16. ABSTRACT

Scanning electron microscopy interfaced with automated image analysis and x-ray energy spectroscopy (SAX) can be employed for individual particle characterization of aerosol samples in a time efficient manner. Such an individual particle analysis partitioning of the bulk chemical data provides a new perspective on the heterogeneity of ambient aerosols. As particles are individuals, it is possible to increase the source resolution of conventional receptor models which are based on bulk chemical analyses. This paper presents a particle class balance (PCB) which is directly analogous, in mathematical approach, to the CMB types of receptor models.

Samples of ambient aerosol and potential source materials from Syracuse, NY were characterized by SAX. Particles from both types of specimens were sorted by a fixed classification algorithm wherein the number percent and weight percent contribution to each of 25 categories were recorded. The resulting "signatures" of the source standards served as the basis for "unmixing" the composite ambient signatures into their fractional components. This was accomplished by both a linear least squares method and a Q-mode factor analysis/vector analysis technique, for comparative purposes. In both cases, sources were apportioned to 12 generic source types - an improvement in resolution over most bulk chemical analysis models. Results were consistent with what is known of area and point source types and strengths in Syracuse.

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Samples of ambient aerosol and potential source materials from Syracuse, N.Y. were characterized by SAX. Particles from both types of specimens were sorted by a fixed classification algorithm wherein the number percent and weight percent contribution to each of 25 categories were recorded. The resulting "signatures" of the source standards served as the basis for "unmixing" the composite ambient signatures into their fractional components. This was accomplished by both a linear least squares method and a Q-mode factor analysis/vector analysis technique, for comparative purposes. In both cases, sources were apportioned to 12 generic source types - an improvement in resolution over most bulk chemical analysis models. Results were consistent with what is known of area and point source types and strengths in Syracuse.

Introduction

Receptor models for aerosol source apportionment may take a variety of forms. Each is based upon a chemical analytical characterization of atmospheric particles, and each may employ one or more mathematical tools in the treatment of the data (or lack of data) for ambient aerosols and their potential source materials. At present three basic types of receptor models are in use: those based on bulk chemical element analyses, models based upon crystalline component identification and quantification, and models which rely on individual aerosol particle class or type characterizations. Organic molecular compound analysis and quantification will no doubt provide a new type of model.

Bulk chemical element composition models such as the chemical element balance (CEB) or chemical mass balance (CMB) are the most commonly used and are usually based on x-ray fluorescence, neutron activation, or atomic absorption spectroscopy analyses. Several general descriptions of bulk chemical analysis receptor models are available. The mathematical data manipulation techniques which are employed with bulk elemental analysis results include linear least squares, multiple regression, ridge regression analysis and factor analysis.

Receptor models based on aerosol characterization other than elemental composition are relatively new and have not been extensively described in the scientific literature. A provenence analysis utilizing the crystalline component composition (by XRD) of urban aerosol in Rapid City, SD, was carried out by Davis. Casuccio and Janocko utilized computer controlled scanning electron microscopy (CCSEM) for an aerosol particle type based receptor model, but did not describe in detail its operation, applicability, or limitations.

We report here an individual aerosol particle characterization model for source apportionment based on particle analysis by scanning electron microscopy with automated image analysis and x-ray energy spectroscopy (SAX). The model structure is equivalent to the CMB except that ambient and source aerosol bulk elemental composition is replaced by the number or mass contribution of particle types in the aerosol samples. Hence, we have termed this approach a particle class balance (PCB) model. A variety of potential source and ambient atmospheric particle samplings for 1980 and 1981 in Syracuse, New York, serve as the basis for the introduction of the PCB receptor model.

Methods and Results

SAX Particle Characterization

All particle analyses were conducted by SEM-XES using an ETEC Autoscan scanning electron microscope coupled to a Kevex 5100C x-ray spectrometer, and interfaced to a LeMont Scientific B10-E image analysis system. Automation of the processes associated with these types of analyses was done using an Interdata 8/16E microprocessor, which also was responsible for subsequent data manipulation. The use of the computerized system made it possible to analyze significant numbers of particles in a reasonable length of time -- 400-600 particles/hour. SAX is performed in three discrete operations: 1) morphological determinations, 2) elemental composition determination, and 3) classification of particles. To arrive at morphological measurements of individual particles, image analysis procedures were imposed upon the sample. A back scattered electron (BSE) image of particles can be resolved into areas of high contrast and low contrast. Such contrast difference is directly proportional to the atomic number of the atoms producing the BSE image. Thus, particles composed of atomic number elements greater than 11 can be distinguished from standard collection filter media. Threshold gray level selection allows for differentiation of particles from filter substrate. The result of this process is a binary image of the particles in question; the particles appear as a white signal, the filter as a black background. Since binary representations of particles are recognizable to a digital computer, control of image analysis can be automated. Figure 1 shows a secondary electron image of 2 aerosol particles, its BSE binary signal, and the diameter analysis of those images.

Through electron beam and sample electron interaction, the elemental composition of the particle being measured can be acquired. With the software package available on the system we used, the electron beam is positioned at the geometric center of the particle image. x-rays are then collected for 3 seconds. The net X-ray counts above the background channels on either side of each x-ray energy spectrum elemental region of interest are recorded for later use along with the lengths of the four (or optionally, 8) diameters determined for the particle image. These measurements constitute the raw data for each particle, and are utilized for particle classification as well as chemical and physical summaries for the populations of particles in each user defined category.

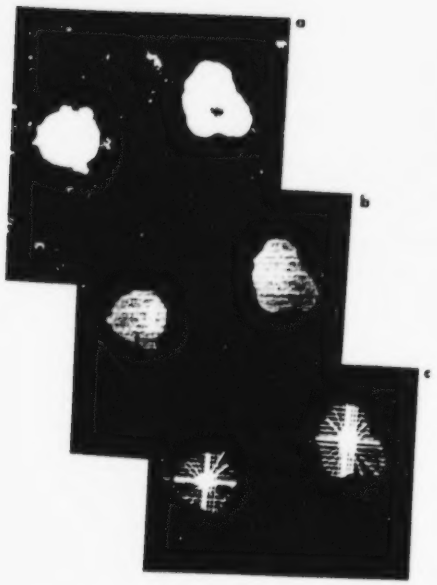

Figure 1. Example of normal secondary electron image of particles at a magnification of 700X (a), their image from the back-scattered electron signal (b), and the LeMont Diameter (Image) analysis of the resultant signal.

Ambient Aerosol Sample Collection

The accuracy and time efficiency of individual particle analysis is quite sensitive to aspects of the aerosol mass loading on the collection substrate. Since samples analyzed in the SEM are generally examined at magnification of 300-1000X, a relatively small field of view (or frame area) results. Typically an area of the filter of 1 to 5 x 10 cm is examined for sample characterization at the optimum loading range. This small area dictates that uniform particle deposition on the filter surface is required to minimize bias in the analysis/sample characterization process.

Aerosol loading on the filters to be subjected to SEM/XES image analysis techniques is important for several reasons. Too high a loading results in overlapping particle images, thus confusing the individual particle characterization. Too light a loading requires excessive operator time in the selection of fields of view for analysis. Particles should be separated from each other about 2-4 m in order to minimize fluorescent x-ray emission from particles adjacent to the one under analysis. We find that these conditions are satisfied by a loading range of 30-80 g/cm of filter surface.

Samples of ambient particulate matter were collected in the Syracuse metropolitan area over a one-year period from May 1980 to May 1981. Small spot samplers were employed for this. Teflon cylinders with a 1 m diameter axial bore were clamped over Nuclepore membranes for sample collection. They were operated either by a small diaphram pump or by a source of bottled vacuum. In either case, samples were generally collected for a 3-6 hr. period depending upon the conditions of atmospheric particle loading. Since the amount of material deposited on the samplers was too little to weigh, the source apportionment results reported represent percentage contributions from the various source types to the average particulate matter loading in Syracuse.

During the complete study period, 60 samples were collected from a variety of locations within the Syracuse air quality maintenance area (AQMA). However, the present results were from one urban residential site, which was sampled over a two-week period at 12-hour intervals. This produced a set of 23 consecutive samples during March and April of 1981.

Characterization of Emission Source Materials

In order to use the source apportionment procedures developed by the PCB model, a source inventory for the study area was conducted. Table I summarizes the number and types of source materials which were characterized for this report. Some of the source standards were not obtained from the Syracuse AQMA, but were felt to be representative of similar materials found there.

Table 1. Syracuse Study Particle Sources

SOURCE TYPE	CHEMICAL PARTICLES	NUMBER OF SAMPLES
Cement Kilns	Ca containing	4
Foundries	Fe and Al-Si materials	9
Chemical Plants*	Ca, Na, Cl, Al-Si cont.	5
Steel Mills*	Fe, Cr, Mn containing	6
Lumber Yard	Ca, K, S containing	2
Plastics Plant	Al-Si containing	2
Refuse Incinerator ♯	Pb, Zn etc. containing	2
Oil Burner ♯	S, V, Al-Si rich	4
Lithophilic Mat. (area source)	Al-Si, Ca, Ti, Fe cont.	9
Automobiles	Pb, Br, Al, Fe containing	2
Wood Burners	Ca, K, S rich	2
Vegetative Debris (area source)	Ca, K, S, organic cont.	1
Secondary Parts. (area source)	S containing	1
		47

* Includes Coal Fly Ash Samples

♯ Obtained from outside the Syracuse AQMA

Bulk-point and-area source standard materials were deposited on membrane filters for analysis by shaking the material in a plastic bag, letting the large (15 m) particles settle for about 10 seconds, and then inserting an air sampler into the bag for 5-15 seconds. This sampling procedure attempts to collect particles of the source material representative of that in the IP range which could be transported from source to receptor site. Replicate samplings of this nature show greater than 85% similarity. In preparation for the SEM analysis, portions of the membrane filters were mounted on carbon stubs using colloidal graphite in isopropanol. The samples were then coated with 20-40nm carbon in a high vacuum evaporator.

Particle Classification

A critical step in the individual particle analysis procedure as applied to the PCB model for source apportionment is the user defined classification of particles on the basis of their measured characteristics. Each category in the chemistry definition file (CDF) is spec-

ified by the operator and saved for future use. The CDF used here contained 25 separate classes of particles based on the observed relative net x-ray contributions for up to 27 elements, as well as a variety of physical parameters (i.e. width/length ratio, avg. diameter, etc.). Details of the classification scheme are given in Johnson, et al.

The individual classes in the CDF were based upon analyzes of standard materials that describe each class. For example, the marine aerosol category was defined by analyzing air samples obtained from a sampling expedition in the Carribean Sea; the wood combustion category was derived by analyzing ash from domestic as well as industrial wood burning operations; the combustion cars category was based on samples from auto exhaust, and so on. Source materials are not pure; in this sense; for instance, some real fly ash might be classified as lithophilic. (a general alumino-silicate type material).

The data collected from a population of discrete particles was summarized in a variety of ways. The weight % in each class was based on an estimate of individual particle weight. Particle volume is computed from the image assuming it to be a prolate spheroid rotated around the major axis. These were broken down into user defined size categories (based upon average particle image diameter). For our work, both numbers % and weight % of the total sample in each chemical category were used as input data for the mathematical apportionment manipulations.

Analytical Reproducibility

Several experiments were conducted to determine the variability of the analytical results obtained from SAX. Similarity coefficients were used as an index since they are sensitive to small differences in the classification results of individual particle analyses. (The similarity coefficient is the sum, over all user-defined categories, of the minimum value that two samples in a pairwise comparison have in common). With skilled operators, and the analysis of 1000 particles each from the same sample, similarity coefficients of 90% were achieved for between-operator comparisons for weight percent by particle class signatures. For single operator replicate analyses of fields from a sample of ambient aerosols, we also have found 90% similarity coefficients. As the number of particles analyzed increases, the similarity of replicates increases in direct accordance with counting statistics.

Real sample variability, as observed from replicate samplings with samplings in the same location, exceeds the analytical uncertainties described above. In two experiments with triplicate samplers whose inlets were placed within 10 cm of each other, an average similarity coefficient of 81% for weight percent histograms was observed for pair-wise comparison. In a third experiment in which 6 spot samplers were deployed synoptically at 30 meter spacings, a mean similarity coefficient of 74% for replicates was observed. In all these tests, the variability resulting from individual particle class differences was observed to be randomly distributed. Thus, we take the over all uncertainty of our analyses to be approximately 25% where we mean to relate particle classification to reproducibility.

Mathematical Apportionment Techniques

For the first SAX data treatment a linear least squares procedure was used to fit the source data to the observed ambient particle class distributions. Due to matrix inversion problems in the programs used, the most efficient number of sources to be fit were 12-17. Both weight and number percent (of the total sample) apportionment vectors were calculated. After obtaining information as described for particle class distributions within the samples, a routine very much similar to the previously mentioned CMB technique was used to arrive at a best-fit of individual source magnitudes to the observed data.

A PCB replaces the elemental distribution of CMB with a particle class distribution generated by SAX. The replacement occurs in the source signature matrix. From this point the same mathematics apply to both PCB and CMB techniques. The steps involve: Calculation of a predicted value for the proportion of Class k for all sources j

$$P = (M)(C) \qquad \text{(Equation 1)}$$

where

P = predicted proportion of class K
C = proportion of class K in source j
M = fractional abundance of source j

and minimization of the value of X with a linear least squares regression that minimizes the value of $(O - P)$ in Equation 2 below:

$$X = (O - P) / K \qquad \text{(Equation 2)}$$

where,

O = observed proportion of class k

= total error term associated with data

The result of PCB is a vector of mathematically correct individual source magnitudes for a given receptor (sampling) location. Mathematically correct does not imply absolutely correct, since a best-fit procedure may project obviously erroneous results (i.e. 100%, or 0%).

Each ambient aerosol sample and specimen of source materials was characterized by SAX under the same SEM operating conditions, and utilizing the same CDF algorithm for particle type sorting. From the base of 47 source signatures an average 12 source type by 17 particle class source signature matrix was created for an unweighted linear least squares fit source apportionment of the 23 ambient aerosol samples collected at the site in Syracuse. This amounted to 11,500 ambient particles analyzed. Since, in the SAX data summary routines, both population percent by particle class and total aerosol weight percent by particle class histograms are produced, least squares apportionment solutions for both types of signatures were calculated. The results, expressed as a fractional mass contribution to the total ambient aerosol sample, are shown in Table II.

Table II. Source apportion for 23 Syracuse aerosol samples collected at one site, March and April, 1981. Results are average wt.% contributions for Q-Model.

PARTICLE SOURCE TYPE	LEAST SQUARES NO.%	LEAST SQUARES WT.%	Q-MODEL WT.%
Organic material	2	0	3
Salt resembling	0	0	2
Secondary sulfate	1	0	0
Ca and S rich	8	5	21
Wood flyash	1	-7	0
Auto emissions	4	2	2
Oil flyash	1	1	3
Coal flyash	12	-17	5
Steel mill emissions	3	7	5
Limestone/calcite	10	12	13
Other Lithophilic	56	89	44
Miscellaneous	3	1	2

Also shown in Table II are the source apportionment results from the PCB model as obtained by a different mathematical treatment of the same SAX data - a factor analysis/vector analysis approach called CAB-

FAC/EXTENDED Q-MODEL as described by Full, et al. For Q-Model, data on total weight percent contribution for each of the 25 particle types were used in the solution as opposed to 17 particle types in the linear least squares solution described above. The weight percent-by-particle type signatures for 47 possible sources and each of the 23 ambient aerosol samples were combined and submitted to a Q-Mode factor analysis which employed an orthogonal (VARIMAX) factor axis rotation. The computer program employed identified the most extreme samples (now in Varimax factor space) as the boundary points defining a hyperellipsoid. The number of such boundary was user specified at 15. Solutions were sought which did not include any of the ambient aerosol samples as end members. The apportionment of the ambient samples was then taken as the vector composition description of the ambient specimens in terms of the selected extreme or end member source samples. The 15 specific end members identified were then collapsed to the 12 generic categories shown in Table II.

Discussion

The chemistry definition file, or sorting algorithm, used for the SAX results in this work may not be optimum for general application as a descriptive tool. Every set of particles collected at a sampling site represents a subset of the particle types which historically might occur there. That is, during a discrete sampling period, not all potential sources would be contributing to the receptor site aerosol. As this phenomenon has geographic variations as well, optimum particle sorting schemes might be different for samples collected in different locations. A sample specific (or variable, tailor made) particle sorting scheme would no doubt be the most cleanly descriptive as a microscopic technique. If accurate specific particle type and/or source identities could be established for each feature observed in the SEM, then an absolute source apportionment would be possible. There are two difficulties in attaining this goal: 1) particle sources are not pure - they generally contain a variety of particle types similar to other sources, and 2) the current state of the art for SAX technology is insufficient for unique feature discernment - uncertainties in both x-ray data acquisition and the feature image analysis lead to uncertainties in classification. To the extent that unique particle types from specific sources can be identified by SAX, then a variable sorting algorithm may be employed. Since sources are variable and complex, such an approach in a PCB receptor model appears to have limited applicability; especially when airshed - specific source information is not available.

We have elected to employ a fixed particle sorting scheme to be uniformly applied to source materials as well as ambient samples. The particle classifications used are diverse and intended to represent a variety of generic source types. We recognize that in application of this method we lose some purely descriptive capabilities of the data summary as well as increase the possibility of particle class overlap when comparing signatures of similar sources, but these disadvantages are outweighed by the advantages of internal or relative sample and source analytical consistency. The signature overlap we experience can be sorted out by a variety of statistical methods.

The source apportionment results for the Syracuse aerosol, as shown in Table II, seem reasonable based on our knowledge of the emission source inventory for the local AQMA. Of course, verification of these results will only be possible by comparison to results of other receptor and dispersion models.

Considering the linear least squares results it seemed, at first, surprising that such differences in average apportionment results for coal flyash and lithophilic materials could result from the use of SAX particle class population percent signatures as compared to the particle class weight percent signatures. However, the least squares generic source signature matrix is an average for a number of specific sources. In the analysis of the ambient samples, a few large particles could distort the signature (or significantly weight it) towards non-average values. Thus the coal fly ash/general lithophilic colinearity shown by a -17% and a + 89% in the least squares weight percent apportionment of Table II might be resolved if the SAX results were size fractionated in the apportionment model. Preliminary results show promise and will be presented as a theoretical approach to size-specific apportionment and source identification in a subsequent publication. SAX procedures make it very easy to peruse size specific results since the image analysis of each individual aerosol particle contains detailed morphological information.

The Q-Model apportionment results shown in column three of Table II are very similar to the number percent results obtained from the least squares solution. The total particle class weight percent contribution signature was employed in the Q-Model calculation. While a few large particles in a given source or ambient sample may also have distorted these results, the Q-Model apportionment was based on a population of 47 specific source signatures, and not directly on generic signatures resulting from averaging several sources.

Conclusions

Individual aerosol particle characterizations by SAX are a sufficient data base for the development of a new type of receptor model for source apportionment. Computer control of SEM/XES functions speeds the acquisition of such data so as to make individual particle analysis an economically feasible technique. Since hundreds of particles can be recovered per hour, large data bases for source and ambient aerosol samples can be assembled. This increases the potential source type resolution of SAX based receptor models by increasing the number of degrees of freedom available for multivariate statistical treatments of the data. SAX receptor models would appear to be most useful in situations where conventional CMB applications have trouble separating similar sources because of multicolinearities. To be sure, SAX results may suffer from colinearities in particle definition, but used in conjunction with other techniques, different perspectives may be obtained. Where unique particle class source tracers are present in an aerosol, receptor models based on particle type classification offer an extremely efficient method for the validation of dispersion models. The automatic size fractionation available through image analysis of SEM signals could provide a new and valuable perspective for receptor based source apportionment models.

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